RI 8480

Bureau of Mines Report of Investigations/1980

A Simple, Low-Cost Method for the Dissolution of Metal and Mineral Samples in Plastic Pressure Vessels

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UNITED STATES DEPARTMENT OF THE INTERIOR

I 28.23:

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UNITED STATES DEPARTMENT OF THE INTERIOR
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BUREAU OF MINES
Lindsay D. Norman, Acting Director

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This publication has been cataloged as follows:

Farrell, Raymond F

A simple, low-cost method for the dissolution of metal and mineral samples in plastic pressure vessels.

(Report of investigations • Bureau of Mines ; 8480)
Bibliography: p. 12-13.
Supt. of Docs. no.: I 28.23:8480.

1. Metallurgical analysis. 2. Solution (Chemistry). I. Matthes, Steven A., joint author. II. Mackie, Alick J., joint author. III. Title. IV. Series: United States. Bureau of Mines. Report of investigations : 8480.

TN23.U43 [QD133] 622s [543'.02] 80-607936

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A SIMPLE, LOW-COST METHOD FOR THE DISSOLUTION OF METAL AND MINERAL SAMPLES IN PLASTIC PRESSURE VESSELS

by

R. F. Farrell,[1] S. A. Matthes,[2] and A. J. Mackie[3]

ABSTRACT

A goal of the U.S. Department of the Interior, Bureau of Mines, is the advancement of minerals technology through the development of improved metals and minerals processing. In support of this goal, the Bureau of Mines has developed a general method for preparing solutions from a wide range of metal and mineral samples for chemical analysis. The development of the method from current knowledge of vessel material, pressure dissolution techniques, and the fluoride-boric acid reaction is given. The method uses 7 ml of a mixture of HCl-HNO_3-HF acids in a ratio of 3.5:2:1.5 sealed in plastic bottles that act as pressure vessels when heated in a boiling water bath. A final volume of 100 ml is obtained by the addition of 93 ml of a 1.5-pct H_3BO_3 solution to the bottles, which are reheated; precipitated fluorides as well as sample residue are dissolved by the fluoride-boric acid reaction. A copper slag and a steelmaking slag treated using this method were analyzed by atomic absorption spectroscopy. yielding values for Si, Mg, Ca, Al, Fe, Mn, Na, As, Pb, Sb, and Zn very close to certified values with an average relative standard deviation of 2.13 pct. The low cost, reduction in preparation time, ease and safety of sample handling, conservation of reagents, and ability to dissolve volatile materials without loss make this method superior to traditional wet digestion techniques for many materials, and ideal for use with atomic absorption, and optical emission or X-ray emission spectroscopy.

INTRODUCTION

The modern analytical laboratory is called upon to analyze a wide variety of inorganic sample types. Because of increasing environmental concerns, the list of routinely analyzed elements continues to grow, and as instrumental techniques of analysis improve, the chemist is asked to analyze for more and more elements in each sample. Advancements in analytical instrumentation, particularly atomic absorption and plasma-emission sprctroscopy, have made the

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preparation time, rather than the analysis time, the limiting factor in chemical analysis. Instrumental analysis has advanced into the space age while sample preparation languishes in the beaker age. The Albany Research Center produces and tests a wide variety of metal and mineral products constitutes a research goals. The determination of elements in these products constitutes a large part of the work of the analytical section of the research center. To expedite these determinations, a simple, low-cost method for the preparation of analytical solutions from a wide variety of inorganic sample types has been developed.

Traditional methods for the preparation of analytical solutions involve acid digestion in open, heated vessels (7-8).[4] Sample type dictates which acid mixture, temperature, and vessel material are required for dissolution. Silicates and other siliceous materials require hydrofluoric acid (HF) for dissolution (10). This prohibits the use of glassware, which is also attacked by HF, and forces the chemist to use costly platinum or Teflon[5] vessels. Other sample types may require a nitric acid (HNO_3), hydrochloric acid (HCl) mixture, unsuitable for platinum ware. Many elements form volatile compounds, such as SiF_4 and $AsCl_3$, which are lost at temperatures necessary for complete dissolution of the sample (10). Thus, if several elements are to be determined in a single sample, more than one dissolution procedure is frequently necessary for the production of a good analytical solution. A chemist faced with the task of analyzing a sample for Pb, As, Bi, Zn, Si, Al, and Ca, may have to perform several different wet digestions in order to produce solutions for the determination of each element. This process requires large amounts of reagents, two or three different types of digestion vessels, and a great deal of time and working space.

Ideally, the chemist would prefer a low-cost, interference-free method whereby the same dissolution vessel could be used in the preparation of each sample, and one acid mixture could serve to dissolve each element in the sample. This ideal system could only be achieved if four criteria were met: (1) The vessel is resistant to attack by an acid combination and a temperature suitable for dissolving every sample, (2) the vessel material contains none of the elements to be analyzed, (3) the vessel prevents volatile elements from escaping, and (4) the vessel cost is not prohibitive for routine use.

The use of closed or sealed dissolution vessels would prevent the loss of volatile compounds during digestion, thus satisfying the third criterion. However, the use of closed vessels adds a new dimension to the problem of finding a suitable vessel material. Because of the temperatures necessary for dissolution, significant vapor pressure is built up by the acids inside the closed vessel. Former investigators (5-7) have solved this problem by heating the dissolution vessels inside a pressure vessel. The differences between the internal and external pressure of the dissolution vessel are thereby reduced (2-3, 5-6, 9, 12-14, 16); however, the analysis cost is

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[4]Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

[5]Reference to specific makes and models of equipment does not imply endorsement by the Bureau of Mines.

increased, and the dissolution procedure is complicated. A more desirable method would use plastic pressure vessels sufficiently resistant to heat, pressure, and acid to enable dissolution to proceed without the use of an exterior pressure chamber.

Materials that have been considered for the dissolution vessel are polyethylene (5), polypropylene (2, 5-6, 12-14), Teflon (2, 5, 18), polycarbonate (5-6), and polysulfone. Glass or metal containers are unsuitable because they could contain elements to be analyzed and are susceptible to HF or HCl-HNO_3 attack. The appropriate physical properties of the possible vessel materials are summarized in table 1.

TABLE 1. - Physical properties of plastics

Plastic	Maximum use temperature, ° C	Transparency	Flexibility	Tensile strength, psi
Polyethylene.....	80	Translucent.	Very flexible	2,000
Linear polyethylene..	120	.....do.....	Rigid.....	4,000
Polypropylene.....	125	.....do.....	.....do.....	4,000
Polymethyl pentene...	175	Clear.....	.....do.....	4,000
Teflon.....	205	Translucent.	Very flexible	3,000
Tefzel.....	180	.....do.....	Flexible.....	6,500
Polyvinyl chloride...	70	Clear.....	Rigid.....	6,500
Polycarbonate.....	135	.....do.....	.....do.....	8,000
Polysulfone.....	170	.....do.....	.....do.....	10,000

Because the vessel must withstand temperatures approaching 100° C for adequate dissolution, polyvinyl chloride and polyethylene are unsuitable. High tensile strength and rigidity are also desired since the plastic must withstand the pressures generated by the volatilization of the acids at this temperature. The Bureau previously reported that polyethylene, polypropylene, and Teflon had insufficient rigidity and tensile strength to maintain vessel shape and to prevent vessel failure (5). Glass or metal bombs lined with Teflon work well but are much more expensive than plastic vessels.

Of the thermoplastics investigated, only polycarbonate and polysulfone have the necessary combined rigidity and tensile strength. Polycarbonate is transparent, strong, and rigid; it is autoclavable, nontoxic, unbreakable, and one of the toughest of the thermoplastics. Polysulfone, which is just now commercially available, has a higher tensile strength than polycarbonate and is more resistant to oxidizing acids and HCl (table 2) and more capable of withstanding higher temperatures than polycarbonate. Some experimental bottles supplied by Nalge Co. have proven to be very satisfactory.

TABLE 2. - Percent tensile strength retained at 95° C (24-hour exposure)

Reagent	Polycarbonate	Polysulfone
10 pct HCl.....	100	100
37 pct HCl.....	0	100
10 pct HNO_3.....	100	100

The dissolution system described previously (5) used sealed polycarbonate bottles for the rapid preparation of solutions from steelmaking slags for analysis by atomic absorption spectroscopy (AAS). This system used a pressure vessel to equalize external and internal pressure on the bottle walls and an 3.5:1.5-ml HCl-HF mixture. The method presented here departs from this system by eliminating the external pressure vessel and improving the acid mixture by adding 2 ml HNO_3, a strong oxidizer.

This new mixture was established by experimenting with various acid combinations. From tables 2 and 3, it can be seen that concentrated HCl, HF, or HNO_3 are not recommended for use in polycarbonate. However, by selecting an appropriate mixture of these acids, the relative percentage of HCl, HF, and HNO_3 can be reduced to a level at which the plastic will not be significantly attacked. Degradation of the polycarbonate can be reduced further by limiting the amount of time the acids are in contact with the plastic.

TABLE 3. - Chemical resistance chart

Acid, percent	Polycarbonate	
	At 20° C	At 50° C
Hydrochloric:		
1-5.....	E	E
20.....	E	G
35.....	G	F
Hydrofluoric:		
4.....	G	F
48.....	N	N
Nitric:		
1-10.....	E	G
50.....	G	F
70.....	F	N
Perchloric 70.....	N	N

Key to Classification Code:

E--30 days of constant exposure cause no damage. Plastic may even tolerate exposure for years.

F--Some signs of attack after 7 days of constant exposure to the reagent.

G--Little or no damage after 30 days of constant exposure to the reagent.

N--Not recommended; noticeable signs of attack occur within minutes to hours after exposure. (However, actual failure might take years.)

Source: Nalge Co.

Elimination of the exterior pressure vessel greatly simplifies the dissolution procedure and reduces the total analysis cost. In both methods, the addition of boric acid (H_3BO_3) to the final solution dissolves precipitated fluorides and sample residue and helps eliminate matrix interferences in AAS analysis.

The use of H_3BO_3 to form a fluoro-boric acid matrix is well documented (2-3, 5-6, 9, 11-14, 17-19). Addition of H_3BO_3 to solutions containing HF or inorganic fluorides produces the two-step exothermic reaction:

$$H_3BO_3 + 3HF \longrightarrow HBF_3OH + 2H_2O,$$

$$HBF_3OH + HF \longrightarrow HBF_4 + H_2O.$$

By this reaction, excess fluoride is combined with the H_3BO_3 producing a stable solution that will not readily attack glass and will keep most cations in solution for weeks (2, 5, 8, 12-14, 16).

Several investigators have reported various interferences present between elements in the fluoro-boric system when using atomic absorption (6, 12-15). The interference on the absorbance of Si, Al, Mg, Ca, and Fe by H_3BO_3 has also been reported. Most methods compensate for these interferences involving the fluoro-boric system by using standards containing the relative amounts of each element and H_3BO_3 contained in the sample solution (3, 9, 12-15). However, other investigators have found the fluoro-boric matrix to be interference-free (2, 5, 11, 17). In some cases, interferences were eliminated by analyzing all elements on the nitrous oxide-acetylene flame (17). Most studies did find that H_3BO_3 should be added to aluminum standards if determinations of this element were made without dilution of the fluoro-boric solutions (5-6, 12-15).

Price (17) states that interferences arise due to incorrect amounts of H_3BO_3 and HF in the fluoro-boric system. If a minimum amount of HF is used for dissolution and just enough H_3BO_3 is added to complex the excess fluoride, correct results are obtained even when samples are not comparable in composition to standards. Interferences arise when an excess of HF is used to dissolve the sample. If too much H_3BO_3 is then added to complex all of the fluoride, the viscosity, evaporation, and atomization rates change. If not enough H_3BO_3 is added to complex the fluoride, some precipitation of fluorides or formation of stable fluoride complexes may hinder the atomization process.

The method presented here seeks to avoid these problems by minimizing the HF used in dissolution and thus reducing the amount of H_3BO_3 necessary for complexing the fluoride.

EXPERIMENTATION

Equipment

1. Shatter box grinder.
2. Boiling and cold water baths.
3. Polycarbonate bottles, 250 ml with polypropylene or Teflon screw caps.
4. Teflon tape.
5. Bottle-holding rack (fig. 1) fabricated from stainless steel or heat-resistant plastic. The plates are 30 cm in diameter, spaced 7.5 cm apart, and the bottle holes are 7.0 cm in diameter.

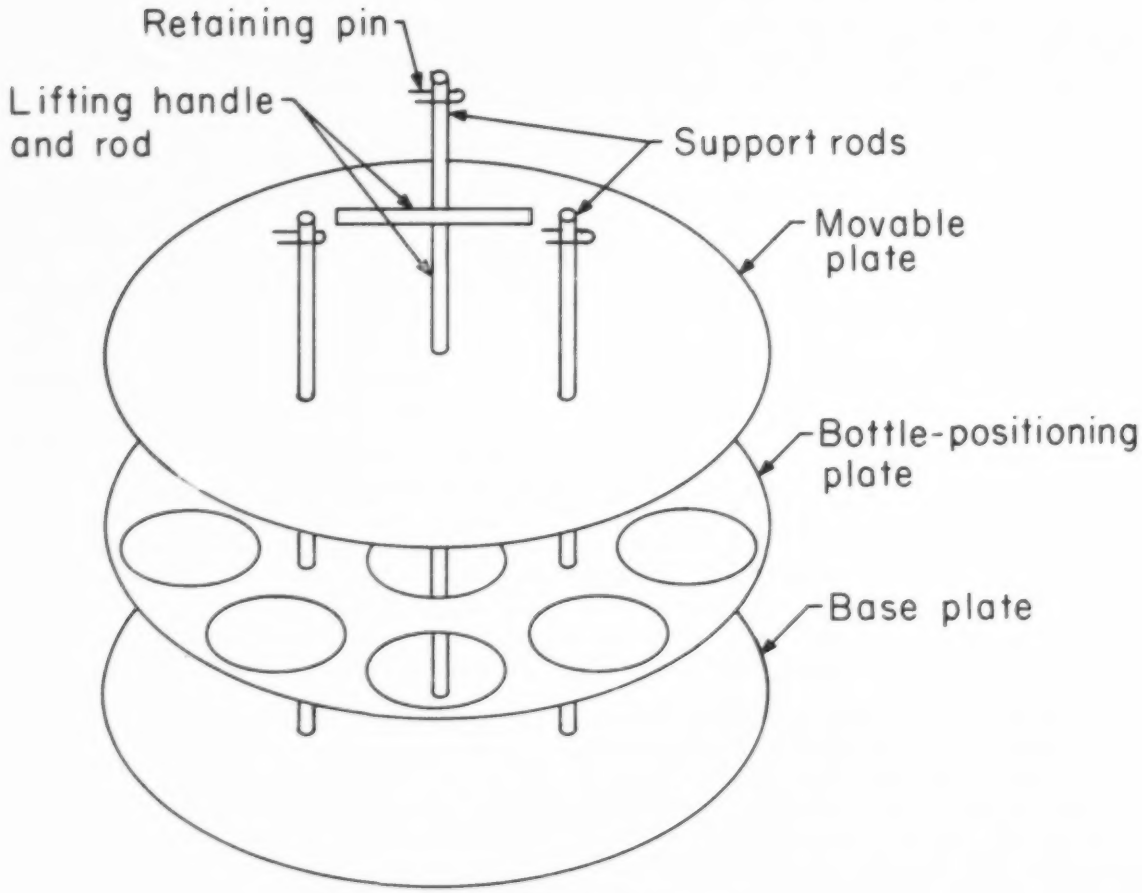

FIGURE 1. - Bottle-holding rack.

Reagents and Solutions

Boric acid solution (1.5 wt-pct per volume) made by mixing 60 grams of high-purity H_3BO_3 crystals (99.99 pct) with 4,000 ml of distilled H_2O.

1. HCl-HF acid mixture (7:3) - made with concentrated reagent-grade acids.

2. HNO_3 - concentrated, reagent-grade acid.

Final acid percentage is given below.[6]

Reagent-grade acid	Actual amount in acid, wt-pct	Relative volume in mix, ml	Relative amount in mix, wt-pct (approx.)
HCl.....	37	7	20
HNO_3.....	70	4	20
HF.....	48	3	10

Pressure Dissolution Apparatus

1. Bottle-holding rack and boiling water bath (fig. 2).

2. Wide-mouthed polycarbonate bottles, 250 ml with screw caps, threads are double-wrapped with Teflon tape (15 cm). Polypropylene screw caps have been used with this method, but Teflon caps are available that give a tighter seal and last through several sets of bottles.

Method

1. Grind all samples to pass 100 mesh.

2. Place 0.100- to 0.500-gram samples into 250-ml polycarbonate bottles.

3. Pipette (using a plastic pipette) a 5-ml 7:3 mixture of HCl and HF into each bottle, then add 2 ml of HNO_3 into each bottle.

4. Screw caps on tightly.

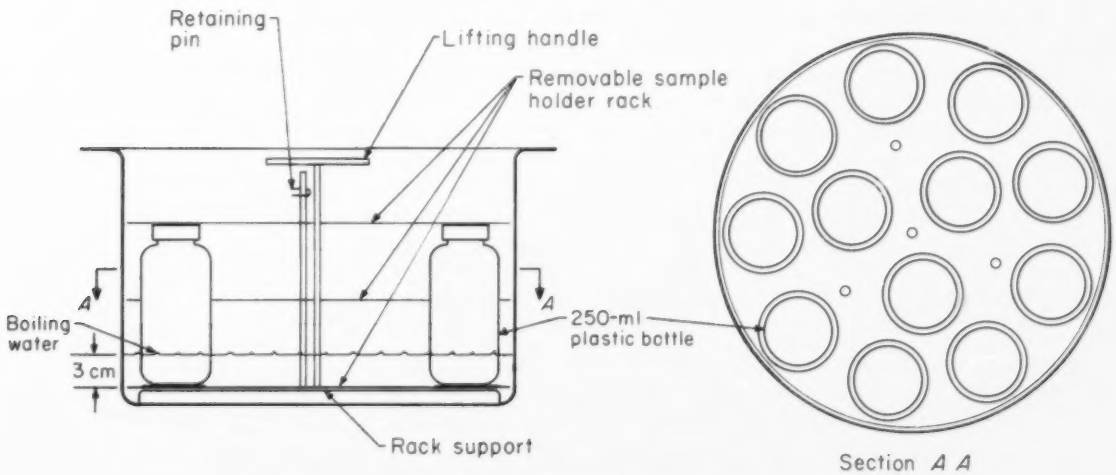

FIGURE 2. - Bottle-holding rack and boiling water bath.

[6]The derivation of these data is explained in the appendix.

5. Put bottles into bottle-holding rack, and place the rack into the boiling water bath for 15 min. (The water level should be about 3 cm above the bottom of the plastic bottles.)

6. Remove rack and bottles and cool in running water for 2 min.

7. When cool, remove caps from the bottles and add 93 ml of a 1.5-pct-H_3BO_3 solution to each bottle using a precision liquid dispenser.

8. Screw caps on tightly.

9. Put bottles into the bottle-holding rack, and place the rack back into the boiling water bath for 15 min.

10. Remove rack and bottles and place in cold water bath. When the final solutions are cool, they are ready for analysis.

RESULTS

The dissolution method presented was used to prepare analytical solutions from a steel slag and a copper reverberatory slag. These solutions or appropriate dilutions were analyzed by flame atomic absorption spectroscopy. Standards were made from dilutions of 1-mg/ml stock solutions and were not matrix corrected (5). Seven elements were determined in the steel slag (Na, Fe, Mg, Ca, Al, Si, Mn); and four were determined in the copper slag (Zn, Pb, As, Sb). These elements were selected because they could be determined by classical wet methods or by open-vessel dissolution followed by atomic absorption only after four separate dissolution techniques. Using pressure dissolution and atomic absorption, a single solution was all that was required.

Ten samples from each material were analyzed, and the average weight-percent and relative standard deviation are given in tables 4 and 5. A sample weight of 0.40 gram was used for the copper slag and 0.25 gram for the steel slag.

TABLE 4. - Analysis of control steel slag, percent

	FeO	MgO	CaO	Al_2O_3	SiO	MnO	Na_2O
Mean.....	13.9	3.15	53.3	1.43	18.5	5.30	0.027
Relative standard deviation....	1.60	1.37	.75	2.22	.83	1.79	5.70
Certified value.....	14.1	3.1	53.7	1.47	18.8	5.2	.028

TABLE 5. - Analysis of control copper slag, percent

	Zn	Pb	As	Sb
Mean.....	2.41	0.96	1.35	0.77
Relative standard deviation...	2.03	1.96	2.44	2.74
Certified value.....	2.46	.96	1.36	.77

From tables 4 and 5, it is apparent that the single sample solution produced by the bottle dissolution method yields AAS results that are in excellent agreement with the certified values. Relative standard deviations ranged from 0.75 pct to 5.70 pct with an average of 2.13 pct. It should be remembered that these values were obtained using elemental standards uncorrected for any matrix effects. Mg, Ca, Al, Si, and As (16) were determined using a nitrous oxide-acetylene flame, and Fe, Mn, Na, Zn, Pb, and Sb were determined using an air-acetylene flame. To correct for ionization effects 0.8 g/l and 4 g/l NaCl were added to sample and standards for Ca and Al, respectively (5). Fe, Mn, Si, Al, Ca, and Sb were run using a 0.2-nm slit, and Mg, Na, Zn, Pb, As using a 0.7-nm slit.

DISCUSSION

Pressure dissolution in plastic vessels has been used in this laboratory for over 3 years. The method presented here has been used successfully for over a year on a wide variety of samples, including steel slags, clays, phosphate rock, copper reverberatory slags, and mattes. It was found that some samples are not entirely dissolved by the method, but were run with success because all of the elements of interest were put into solution. However, if a residue is present and suspected of being an insoluble fluoride or some precipitated substance, further heating of the final solution may be required to achieve dissolution.

Although the sealed bottles prevent significant loss of volatile substances from the samples, some loss of NO_2 produced by acid decomposition may result. A gradual yellowing of the plastic will occur as more and more NO_2 (4) diffuses into the walls of the vessel over several runs. In addition, gas may diffuse through the plastic walls or escape around the screw cap.

NO_2 and HF are toxic (4) and affect the lungs and respiratory tract. Therefore, the entire dissolution process should be carried out in a laboratory hood.[7]

Because of the reaction of the acid with the sample and subsequent evolution of NO_2, a reduction of the volume of the acid may occur. With a small sample size this change is negligible, and even with a 0.5-gram sample the volume loss due to this effect is less than 1 ml. Consequently, after the addition of the H_3BO_3 solution, the final volume is reduced by less than 1 pct.

Using the method, a copper reverberatory slag was analyzed for arsenic, and a Brammer steel slag was analyzed for silicon. Values of 1.35 pct arsenic and 18.5 silica compared favorably to certified values of 1.36 pct and 18.8 pct for arsenic and silica, respectively. This comparison demonstrated that the loss of volatile compounds of arsenic and silicon is negligible.

[7]The "maximal acceptable concentration" for NO_2 is given (4) as 5 ppm (parts per million by volume) for continuous exposure during an 8-hour working day and as 25 ppm for 3 to 5 min. The odor threshold is about 5 ppm; the visual threshold for the brown NO_2 fumes is 75 to 100 ppm.

If AAS determinations must be made by aspirating the sample directly from the bottle in order to determine elements at low concentration, standards should be used that also contain 1-1/2 pct H_3BO_3. This precaution is not necessary for most elements, but the different viscosities, vaporization, evaporation, and atomization rates of the fluoro-boric system may cause some deviation in observed absorbance if compared against standards that do not include boric acid. This effect is most noticeable with Aluminum (5); however, further dilution of the sample with water eliminates the problem.

CONCLUSIONS

The pressurized plastic bottle dissolution method is a radical departure from the traditional open-vessel technique as a general method of solution preparation and has many advantages over traditional methods:

1. Use of a high tensile-strength thermoplastic allows the combined use of HF, HCl, and HNO_3 simultaneously under pressure in a closed system.

2. Use of a closed pressure vessel permits the digestion to be carried out at a higher temperature than in an open vessel, thereby speeding up the dissolution process and helping dissolve difficult samples.

3. Volatile compounds such as SiF_4 and $AsCl_3$ are not lost.

4. The fluoro-boric acid reaction keeps most elements in solution, including the troublesome elements, Si, Pb, Sb, Bi, Mo, Al, Se, and Te. The H_3BO_3 tends to eliminate matrix effects and inter-element interference for instrumental analysis, allowing the use of single-element standard solutions for most elements.

5. One dissolution procedure produces a solution from which all elements in the sample are analyzed.

6. Plastic vessels will not contaminate the analytical solution with Si, Mg, Ca, Al, or Na.

7. Little space is required and few pieces of equipment are needed for the pressurized dissolution method.

8. The reaction is contained in a closed vessel, making a cleaner, neater, safer reaction with a minimum of fuming acids.

9. Solutions are of an exact volume, eliminating the need of extra volumetric glassware.

10. For most dissolutions, the need for filtration is eliminated.

11. All samples are dissolved using an identical procedure, making an automated system possible.

12. Vessels are light, transparent, unbreakable, disposable, and possibly recyclable (1). The cost of the method is approximately $2.60 per bottle for a minimum eight runs or less than 35 cents per dissolution. If five elements are analyzed per run, the bottle cost is less than 7 cents per determination. The cost is much less than the cost of additional acid necessary for open-vessel dissolution, not to mention the savings on preparation time and savings in money by replacing expensive Teflon and platinum with a cheaper plastic.

13. Combined with atomic absorption spectrophotometry, analyses of samples are much more rapid. Twelve samples can be analyzed for 12 elements in less than 1 work day. This includes weighing, dissolution, and analysis. By conventional methods, this would take at least 4 work days.

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APPENDIX.--CALCULATIONS FOR ACID MIXTURE

The actual volume of acid mixture used to dissolve the samples was as follows, in milliliters:

$$\begin{aligned} HCl &- 3\tfrac{1}{2} \\ HNO_3 &- 2 \\ HF &- 1\tfrac{1}{2} \\ &\overline{7} \end{aligned}$$

To simplify, each volume was multiplied by 2, yielding the following relative volume for the acid mix, in milliliters:

$$\begin{aligned} HCl &- 7 \\ HNO_3 &- 4 \\ HF &- 3 \\ &\overline{14} \end{aligned}$$

The weight-percent of each acid, which is significant in terms of the deterioration of the plastic bottles, was calculated as follows:

$7/14 \times 37 \cong 20$ wt-pct HCl,

$4/14 \times 70 \cong 20$ wt-pct HNO_3, and

$3/14 \times 48 \cong 10$ wt-pct HF.

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